



**Faculty of Manufacturing Engineering**

**EFFECT OF CHEMICAL SURFACE TREATMENT OF PVD TiN  
COATED WC ON THE COATING ADHESION**

**Khairul Izani bin Mohd Zukee**

**Master of Science in Manufacturing Engineering**

**2014**

**EFFECT OF CHEMICAL SURFACE TREATMENT OF PVD TiN COATED WC  
ON THE COATING ADHESION**

**KHAIRUL IZANI BIN MOHD ZUKEE**

**A thesis submitted  
In fulfillment of the requirements for the degree of Master of Science  
in Manufacturing Engineering**

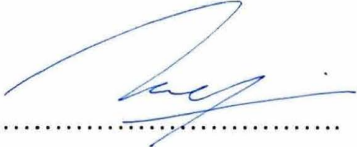
**Faculty of Manufacturing Engineering**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2014**

## DECLARATION

I declare that this thesis entitle “Effect of Chemical Surface Treatment of PVD TiN Coated WC on the Coating Adhesion” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.


Signature :  .....

Name : KHAIRUL IZANI BIN MOHD ZUKEE .....

Date : 24/09/2014 .....

## APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :   
Supervisor Name : Md Nizam Abd Rahman  
Date : 24/09/2014

## **DEDICATION**

To my beloved parents Mohd Zukee bin Mohd Ragam and Nurlina.

Dedicated to my beloved brothers and sister.

Dedicated to all my family and my friends.

Thank you for your support and encouragement.

You all are everything for me.

May Allah bless all of us. InsyaAllah.

## ABSTRACT

Substrate surface treatment prior to the coating deposition is very important in order to obtain good coating adhesion. There are various substrate treatment methods such as polishing, grinding, water peening, and chemical treatment carried out in order to modify the surface characteristics of the substrate. There are quite abundant of research have been done by previous researchers on the substrate surface treatments and one of the methods used was chemical treatment using alkaline and acid solution which was typically performed on tungsten carbide (WC) prior to the deposition of diamond coatings by chemical vapour deposition (CVD). However, study on the effect of substrate chemical treatment using alkaline and acid solution on WC prior to the PVD TiN coating deposition on the coating adhesion is lacking. Adhesion between deposited coatings and substrate is one of the most important success criteria of coating process. The aim of this research is to determine the effect of substrate chemical treatment method using ultrasonic cleaner on the adhesion of TiN PVD coated tungsten carbide. The input parameters evaluated were types of solutions and treatment time. While, the output responses evaluated were surface energy, cobalt content, surface roughness, and adhesion. The characterizations were carried out using sessile drop, SEM/EDX, AFM and Rockwell indenter. The experimental approach adopted was general factorial design. Minitab version 16 software was utilized to analyze the collected data. The chemical treatment using acid for 20 minutes was found to improve the coating adhesion due to formation of uniform peaks and valleys surface morphology. It was found that the coating adhesion was dependent on both surface morphology and surface roughness of the substrate. The cobalt content on the surface layer seems to affect the coating adhesion as well. It was concluded that the substrate surface characteristics were closely related to the performance of the TiN coated inserts.

## ABSTRAK

*Rawatan permukaan substrat sebelum proses pemendapan salutan adalah sangat penting untuk mendapatkan lekatan yang baik. Terdapat pelbagai kaedah rawatan substrat seperti penggilapan, pencanaian, penembakan air dan rawatan kimia yang dijalankan untuk mengubah ciri-ciri permukaan substrat sebelum proses pemendapan salutan. Terdapat banyak kajian yang dijalankan sebelum ini terhadap rawatan permukaan substrat dan salah satu kaedah yang digunakan adalah rawatan kimia menggunakan larutan alkali dan asid yang biasanya dilakukan pada tungsten karbida (WC) sebelum pemendapan salutan berlian menggunakan deposisi wap kimia (CVD). Walau bagaimanapun, kajian tentang kesan rawatan kimia terhadap substrat menggunakan larutan alkali dan asid pada substrat tungsten karbida sebelum deposisi wap fizikal (PVD) salutan titanium nitrida terhadap kekuatan salutan adalah kurang. Kekuatan lekatan antara salutan dan substrat merupakan salah satu kriteria penting dalam menentukan kualiti salutan tersebut. Tujuan kajian ini adalah untuk menentukan kesan kaedah rawatan kimia terhadap substrat tungsten karbida menggunakan pembersih ultrasonik terhadap kekuatan lekatan salutan titanium nitrida. Input parameter yang dinilai adalah jenis larutan dan tempoh rawatan. Manakala output parameter yang dinilai adalah tenaga permukaan, kandungan kobalt, kekasaran permukaan, dan kekuatan lekatan. Analisa pencirian telah dijalankan menggunakan titisan sessile, SEM/EDX, AFM, dan indentasi Rockwell. Pendekatan eksperimen yang digunakan adalah faktorial umum menggunakan perisian Minitab versi 16 untuk menganalisis data yang diperolehi. Rawatan kimia menggunakan asid selama 20 minit didapati meningkatkan lekatan salutan disebabkan pembentukan puncak dan lembah yang sekata. Lekatan salutan didapati bergantung kepada morfologi permukaan dan kekasaran permukaan substrat. Kandungan kobalt pada permukaan substrat juga didapati mempengaruhi kekuatan lekatan salutan. Sebagai kesimpulannya, ciri-ciri permukaan substrat mempengaruhi prestasi salutan titanium nitride ke atas tungsten karbida.*



## ACKNOWLEDGEMENTS

Alhamdulillah and thank you to Allah S.W.T. for giving me strength and ability to accomplish this project research successfully. I would like to take the greatest opportunity to express my sincere and gratitude to my supervisor, Associate Prof. Dr. Md. Nizam bin Abd. Rahman who always give me supports and guidance in completing this research. Many thanks also to my co-supervisor Mr. Mohd Fairuz bin Dimin for his advice and suggestion upon the completion of my thesis. Besides, thanks to all the lecturers and staff of the Faculty of Manufacturing Engineering. Special thanks are also given to the Ministry of Higher Education for support and funding this project through the fundamental research grant scheme (FRGS/2010/FKP/SG02/8 -F00110).

Also to all my fellow friends who involves direct or indirectly that always stand strong beside me in giving opinions and supports throughout our relationship, I really thankful and appreciate it very much.

Last but not least, I would like to express the greatest thanks to my beloved parents and family who always pray and give the encouragement while pursuing my research and project. Their sacrifices are never being forgotten.



## TABLE OF CONTENTS

	PAGE
DECLARATION	
APPROVAL	
DEDICATION	
ABSTRACT	i
ABSTRAK	ii
ACKNOWLEDGEMENT	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vi
LIST OF FIGURES	viii
LIST OF APPENDICES	xi
LIST OF ABBREVIATIONS	xii
LIST OF PUBLICATIONS	xiv
 CHAPTER	
1. INTRODUCTION	1
1.1 Background	1
1.2 Problem Statement	2
1.3 Objectives of Study	3
1.4 Scopes of Study	4
1.5 Significance of Research	4
1.6 Thesis Layout	5
 2. LITERATURE REVIEW	6
2.1 Coating Overview	6
2.2 Physical Vapour Deposition (PVD)	7
2.2.1 Sputtering	7
2.2.2 Unbalanced Magnetron Sputtering	9
2.3 Tungsten Carbide (WC)	10
2.4 Titanium Nitride (TiN) Coating	12
2.5 Coating Adhesion	12
2.5.1 Surface Energy	14
2.5.2 Relationship between Surface Energy and Adhesion	15
2.5.3 Owens-Wendt Method	16
2.6 Importance of Surface Treatment	17
2.6.1 Mechanical Treatment	18
2.6.1.1 Grit Blasting	18
2.6.1.2 Microblasting	20
2.6.1.3 Water Peening	24
2.6.1.4 Grinding	27
2.6.1.5 Polishing	31
2.7 Chemical Treatment	34
2.8 Adhesion Study Characterization Techniques	37
2.8.1 Elemental Composition Analysis Technique	38
2.8.2 Contact Angle Analysis Technique	38
2.8.3 Surface Roughness Analysis Technique	39
2.8.4 Surface Morphology Analysis Technique	40
2.8.5 Adhesion Analysis Technique	40
2.9 Design of Experiment	43

2.9.1 Factorial Design	44
2.9.2 Application of Factorial Design	44
2.10 Summary	45
<b>3. METHODOLOGY</b>	<b>46</b>
3.1 Methodology	46
3.2 Experimental Matrix Design	47
3.3 Substrate Material	48
3.4 Substrate Characterization before Chemical Treatment	49
3.4.1 Sessile Drop Technique	50
3.4.2 SEM-EDX Characterization	50
3.4.3 AFM Characterization	51
3.5 Chemical Surface Treatment	52
3.6 Substrate Cleaning	54
3.7 Substrate Characterization after Chemical Treatment	54
3.8 Coating Deposition	54
3.8.1 Substrate Ion Cleaning	55
3.8.2 Interlayer Coating	56
3.8.3 TiN Coating Deposition	56
3.9 Coating Characterization Methods	57
3.9.1 Rockwell Indenter	57
3.10 Data Analysis	58
<b>4. RESULTS AND DISCUSSION</b>	<b>59</b>
4.1 Introduction	59
4.1 Substrate Characterization before Chemical Treatment Results	60
4.2.1 Surface Energy	60
4.2.2 Cobalt Content	61
4.2.3 Surface Roughness	63
4.3 Surface Pretreatment Result	64
4.3.1 Surface Roughness Analysis	65
4.3.1.1 Surface Energy Analysis	69
4.3.2 Cobalt Content Analysis	73
4.3.4 Coating Adhesion Analysis	76
4.3.4.1 Effect of Surface Energy on Adhesion Strength	86
4.3.4.2 Effect of Surface Roughness on Adhesion Strength	87
4.3.4.3 Effect of Cobalt Presence on Adhesion Strength	88
<b>5. CONCLUSION AND RECOMMENDATION</b>	<b>90</b>
5.1 Conclusion	90
5.2 Recommendation	91
<b>REFERENCES</b>	<b>92</b>
<b>APPENDIX A</b>	<b>109</b>
<b>APPENDIX B</b>	<b>117</b>

## LIST OF TABLES

TABLE	TITLE	PAGE
2.1	List of various tungsten carbide cutting tools based on %Co content	11
2.2	Properties of tungsten carbide (Kalpakjian, 2006)	11
2.3	Forces at the interface of the material (Petrie, 2007)	13
2.4	Surface tension components for water and methylene iodide (Wu, 1982)	16
2.5	Previous research by other researchers on the evaluated process parameters for grit blasting	19
2.6	Previous research by other researchers on the evaluated process parameters for microblasting	23
2.7	Previous research by other researchers on the evaluated process parameters for water peening	26
2.8	Previous research by other researchers on the evaluated process parameters for grinding	30
2.9	Previous research by other researchers on the evaluated process parameters for polishing	33
2.10	Previous research by other researchers on the evaluated process parameters for chemical treatment	35
2.11	Characterization techniques carried out by researchers	37
2.12	EDX characterization techniques carried out by researchers	38
2.13	Rockwell-C characterization techniques performed by researchers using slopes of indentation load versus crack diameter	41
2.14	Experimental works of factorial design	45
3.1	Factors and levels selected for the experiments	48
3.2	General factorial design models	48
3.3	Sumitomo cutting tool dimensions	49
3.4	SEM/EDX parameters setting	51
3.5	Composition of acids solution for surface treatment (Ebnesajjad, 2006)	53
3.6	Ultrasonic cleaner parameters setting	53
3.7	Ion cleaning parameters setting	56
3.8	Coefficient of thermal expansion properties	56
3.9	Interlayer coating parameters setting	56

3.10	TiN coating parameters setting	56
4.1	Data for contact angle and surface energy of as-received samples	60
4.2	Cobalt content of as-received samples	62
4.3	Surface roughness of as-received samples	63
4.4	Experimental results after surface treatment	65
4.5	Surface roughness of substrate after surface treatment	66
4.6	ANOVA for surface roughness	67
4.7	Data for contact angle and surface energy of treated samples	69
4.8	ANOVA for surface energy	70
4.9	Amount of cobalt on surfaces after surface treatment	73
4.10	ANOVA for cobalt content	74
4.11	Crack diameter for substrate (run 1) treated using alkaline solution for 10 minutes	77
4.12	Crack diameter for substrate (run 5) treated using alkaline solution for 10 minutes	77
4.13	Crack diameter for substrate (run 8) treated using alkaline solution for 20 minutes	78
4.14	Crack diameter for substrate (run 4) treated using alkaline solution for 20 minutes	79
4.15	Crack diameter for substrate (run 2) treated using acid solution for 10 minutes	79
4.16	Crack diameter for substrate (run 6) treated using acid solution for 10 minutes	80
4.17	Crack diameter for substrate (run 3) treated using acid solution for 20 minutes	81
4.18	Crack diameter for substrate (run 7) treated using acid solution for 20 minutes	81
4.19	Crack diameter and adhesion slope of substrate after Rockwell indentation	82
4.20	ANOVA for adhesion slope	82
4.21	Summary of before and after surface treatment responses	85



## LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Sputtering process (Brown, 2011)	8
2.2	Planar magnetron cathode (Ray, 1997)	9
2.3	Unbalanced magnetron sputtering system (Kelly and Arnell, 2000)	10
2.4	Description of a wetting state (Young, 1805)	14
2.5	Relation between energy of adhesion and surface energy (Lugscheider and Bobzin, 2001)	15
2.6	Stress modification by microblasting and water peening (Tönshoff and Seegers, 2000)	20
2.7	Effects of substrate treatment methods on cemented carbide superficial structure and on film adhesion (Bouzakis <i>et al.</i> , 2005)	21
2.8	Rockwell indentation test on coated inserts (Bouzakis <i>et al.</i> , 2010)	22
2.9	Critical scratch load as a function of Co content (Kim <i>et al.</i> , 2003)	24
2.10	Influence of water peening pressure on surface composition (Tönshoff <i>et al.</i> , 1999)	25
2.11	Surface of water peened of uncoated and coated substrate (Tönshoff and Blawit, 1997)	25
2.12	Rockwell test and scratch test of water peened sample (Tönshoff and Blawit, 1997)	26
2.13	Tool life of treated substrates (Tönshoff and Blawit, 1997)	26
2.14	Critical load of TiN coatings in function of substrate surface roughness (Kakaš <i>et al.</i> , 2011)	28
2.15	AFM images of ground samples using (a) 400 grit and (b) 2000 grit sand paper (Kakaš <i>et al.</i> , 2011)	28
2.16	AFM images of superalloy substrate surface (Li <i>et al.</i> , 2011)	29
2.17	AFM images of WC sample with various surface roughness (a) 6.3 nm and (b) 7.0 nm (Kim <i>et al.</i> , 2003)	29
2.18	Critical scratch load as function of average surface roughness (Kim <i>et al.</i> , 2003)	30
2.19	The critical loads of the coatings deposited on different roughness substrates (Huang <i>et al.</i> , 2011)	32

2.20	Summary on the effect of surface treatment methods on the coating adhesion	36
2.21	Principle of scratch test (Lacombe, 2006)	42
2.22	Appearances of adhesion failures defined by critical load (a) $L_{c1}$ and (b) $L_{c2}$ (Gerth and Wiklund, 2008)	42
3.1	Methodology flow chart	46
3.2	Tungsten carbide cutting tool insert	49
3.3	Contact angle measurement of liquid droplet	50
3.4	ZEISS EVO 50 SEM	51
3.5	SII SPI3800N scanning probe microscopy	52
3.6	Fisher Accumet Basic AB15 pH meter	52
3.7	JAC-1505 ultrasonic cleaner	53
3.8	VACTEC Korea (VTC PVD-1000)	54
3.9	Top view of magnetron sputtering system schematic diagram	55
3.10	Crack diameter measurement using optical microscope	57
3.11	Adhesion slope measurement plot at three different loads	58
3.12	Mitutoyo HR 500 Rockwell indentation tester	58
4.1	Contact angle of deposited liquid on as-received WC	60
4.2	Normal probability plot of surface energy	61
4.3	Normal probability plot of cobalt content	62
4.4	AFM image of as-received sample	63
4.5	Normal probability plot of surface roughness	64
4.6	AFM images of substrate roughness cleaned using (a) alkaline solution for 10 minutes, (b) alkaline solution for 20 minutes, (c) acid solution for 10 minutes, (d) acid solutions for 20 minutes	66
4.7	Main effects plot for surface roughness	67
4.8	Interaction plot for surface roughness	69
4.9	Main effects plot for surface energy	71
4.10	Interaction plot for surface energy	72
4.11	EDX spectrum of etched tungsten carbide in (a) alkaline solution for 10 minutes, (b) alkaline solution for 20 minutes, (c) acid solution for 10 minutes and (d) acid solution for 20 minutes	73
4.12	Main effects plot for Co content	75
4.13	Interaction plot for cobalt content	76
4.14	Adhesion slope measurement for substrate (run 1) treated using alkaline solution for 10 minutes	77
4.15	Adhesion slope measurement for substrate (run 5) treated using alkaline solution for 10 minutes	78



4.16	Adhesion slope measurement for substrate (run 8) treated using alkaline solution for 20 minutes	78
4.17	Adhesion slope measurement for substrate (run 4) treated using alkaline solution for 20 minutes	79
4.18	Adhesion slope measurement for substrate (run 2) treated using acid solution for 10 minutes	80
4.19	Adhesion slope measurement for substrate (run 6) treated using acid solution for 10 minutes	80
4.20	Adhesion slope measurement for substrate (run 3) treated using acid solution for 20 minutes	81
4.21	Adhesion slope measurement for substrate (run 7) treated using acid solution for 20 minutes	82
4.22	Main effects plot for adhesion slope	83
4.23	Effect of indentation load for alkaline solutions versus time on the crack diameter	84
4.24	Effect of indentation load for acid solutions versus time on the crack diameter	85
4.25	Surface energy before and after treatment and the resulted adhesion strength	86
4.26	Surface roughness before and after treatment and the resulted adhesion strength	87
4.27	Cobalt weight percentage before and after treatment and the resulted and adhesion strength	89

## LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Contact angles measurement after treatment	109
B	Calculation of surface energy	117

## LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
Al <sub>2</sub> O <sub>3</sub>	-	Aluminium oxide
ANOVA	-	Analysis of variance
Ar	-	Argon
C	-	Carbon
Co	-	Cobalt
Cr	-	Chromium
CVD	-	Chemical vapour deposition
DC	-	Direct current
DI	-	Deionized water
DOE	-	Design of experiment
EDX	-	Energy dispersive X-ray
L <sub>c</sub>	-	Critical load
N <sub>2</sub>	-	Nitrogen
OFAT	-	One factor at a time
OM	-	Optical microscope
PVD	-	Physical vapour Deposition
R <sup>2</sup>	-	Coefficient of determination
RF	-	Radio frequency
RSM	-	Response surface methodology
SEM	-	Scanning electron microscopy
TiAlN	-	Titanium aluminium nitride
Ti	-	Titanium
TiN	-	Titanium nitride
VDI	-	Verein Deutscher Ingenieure
W	-	Tungsten
WC	-	Tungsten Carbide

XRD	-	X-ray diffraction
$\gamma$	-	Surface energy
$\gamma_s$	-	Surface tension of solid
$\gamma_l$	-	Surface tension of liquid
$\gamma_{sl}$	-	Interfacial tension of solid/liquid
$\gamma^p$	-	Polar component of surface tension
$\gamma^d$	-	Dispersion component of surface tension

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Nizam, A. R. M., Fairuz, D. M., Rizal, S. M., Warikh, A. B. M., Izani, M. Z. K., Mazliah, M., 2012. A Review of Recent Developments on Substrate Preparation Methods. *Proceeding of International Conference on Design and Concurrent Engineering (iDECON 2012)*, 15-16 October. Melaka: Universiti Teknikal Malaysia Melaka (UTeM).

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Nizam, A.R., Izani, M.Z., Fairuz, D., and Mazliah, M., 2013. Effect of Substrate Chemical Surface Treatment on Surface Energy and the Influence towards Coating Adhesion. *Journal of Advanced Manufacturing Engineering*, 7(2), pp. 29-41.

# CHAPTER 1

## INTRODUCTION

### 1.1 Background

In today's modern manufacturing environment, there is a continually expanding interest for cutting tools within the last phases of manufacturing operations (Gregor *et al.*, 2006). Tool coatings technology had been exploited commercially since 1980s in cutting tool application due to its ability to improve the surface properties of the cutting tool while maintaining its bulk properties (Kim *et al.*, 2003). The application of thin film coatings on cutting tools is to prolong the cutting tool life by improving the surface properties of the tool (Tshinjan *et al.*, 2012; Haubner *et al.*, 2002; Dobrzański *et al.*, 2006). The improved performance of coated cutting tools has been proven and documented. Some of the published works that support this claim are Kim *et al.*, (2003); Nizam *et al.*, (2010); Viana and Machado, (2009); Chokwatvikul *et al.*, (2011). According to Czelle and Barimani (1995), coating on cutting tool, prevents the contact between the cutting tool base material and workpiece, thereby increasing the life of the tools. Research done by Dobrzański and Żukowska (2007) indicated that coated tool lasted twenty times longer than uncoated tool.

One of the common techniques used to deposit hard coatings on cutting tool is physical vapor deposition (PVD). The utilization of PVD coatings in tribological applications comes to be more across the board. Titanium nitride (TiN) coatings is one of the most used and studied material due to its high hardness (Kim *et al.*, 2009; Jaya *et al.*, 2013), highly resistant to corrosion (Kim *et al.*, 2012), and exhibits low wear rate (Yang *et al.*, 2000; Jaya *et al.*, 2013).



One of the important success criteria of coating process is the adhesion between the deposited coatings and the cutting tools (Viana and Machado, 2009; Kakaš *et al.*, 2011; Sonoda *et al.*, 2001). The adhesion strength is influenced by the coating process parameters. In spite of optimized coating parameters, deposited PVD coatings failed because of insufficient surface properties of the substrate. In order to modify the surface properties, the substrate surface treatment is very important prior to coating deposition. There were various surface treatment methods studied by the previous researchers as reported in the literature including mechanical and chemical treatment.

Extensive research has been carried out by previous researchers on those surface treatment methods to determine their effect on the coating adhesion. In these studies, chemical treatment is mostly used for the removal of the Co content and the modification of the surface properties mainly for the deposition of diamond coatings using CVD deposition on WC (Norafifah *et al.*, 2013; Sarangi *et al.*, 2008; Lu *et al.*, 2006; Zhang *et al.*, 2000; Buck and Deuerler, 1998; Deuerler *et al.*, 1996). However, there is lack of study on the effect chemical treatment on the tungsten carbide (WC) prior to the deposition of TiN coating using PVD machine. In addressing this gap, this study look into the effect of various chemical treatment parameters (types of solution and treatment time) on the coating adhesion of PVD TiN coated WC substrate.

## **1.2 Problem Statement**

Coatings must be firmly adhered to the substrate to perform its functions. Therefore, proper adhesion strength must be achieved. This will definitely enhanced the life of the coated material as the coating failures can be minimized. Substrate surface treatment is an important stage affecting the performance of coating adhesion (Carreras *et al.*, 2003). Coating adhesion on substrate can be influenced by various factors including the

substrate surface treatment methods (Bouzakis *et al.*, 2010; Tönshoff and Mohlfeld, 1998; Ahn *et al.*, 2007; Huang *et al.*, 2011) and coating materials (Jakubeczyova *et al.*, 2011; Lugscheider and Bobzin, 2001; Kong *et al.*, 2008). There are quite abundant of research have been done by previous researchers on the substrate surface treatments and one of method have been used was chemical treatment using alkaline and acid solution which is typically performed on WC prior to the deposition of diamond coatings using chemical vapour deposition (CVD). However, study on the effect of substrate chemical treatment using alkaline and acid solution on WC prior to the PVD TiN coating deposition is still lacking. The relationship between coatings adhesion with other process parameters such as solutions (alkaline and acid) and time is unknown. The main aim of this research is to study the influence of those parameters on the coating adhesion of TiN coating deposited on the WC substrate using PVD.

### **1.3 Objectives of Study**

The main aim of this research is to determine the effect of chemical surface treatment on coating adhesion. The specific objectives of this study are:

- i. To study the effect of alkaline and acid solutions during chemical treatment on coating adhesion.
- ii. To identify the effect of chemical treatment time on the coating adhesion.
- iii. To explain the effect of chemical treatment with respect to surface energy, Co content and surface roughness.

## **1.4 Scopes of Study**

This research focused primarily on the effect of chemical surface treatment on the coating adhesion using alkaline and acid solutions. The coating material and substrate materials used in this research were TiN and tungsten carbide (WC) respectively. In this research, the substrate went through the surface treatment process using ultrasonic cleaning machine. The types of solutions used and time were the independent process variables evaluated. Output responses evaluated were surface roughness, surface energy, cobalt contents, and coating adhesion. The collected data were analyzed using the Minitab version 16 software.

## **1.5 Significance of Research**

Chemical surface treatment in modifying surface properties has generated interest in understanding the behavior of alkaline and acid solutions. Mechanical treatments during pretreatment on tungsten carbide are well documented. However, very little has reported on the effect of chemical treatment solutions during pretreatment prior to PVD coating. It is hoped that comprehensive study on this research will enrich knowledge and understanding of chemical surface treatment parameters. This study also expects that chemical surface treatment could improve surface properties of substrate which is potential to save considerable cost through the improvement of the tool life of the cutting tool due to low wear rate. Thus, through the improvement of tool life, the reduction in tooling cost is expected as well. Hence, the machining time for each process could be reduced as well and increased in productivity could be attained.

## **1.6 Thesis Layout**

This thesis consists of five main chapters. It emphasizes on the effect of substrate surface treatment methods on the coating adhesion. First chapter describes about the background of study, problem statement, objectives, scope and the significance of the research. Chapter 2 explains about the comprehensive literature review related to the study which includes the fundamentals of the coating technologies, deposition process, coating adhesion, substrate surface treatments, characterization methods, and design of experiment. Chapter 3 focuses on detailed experimental procedures for this study. It includes the substrate chemical treatment method, coating deposition machine details, and characterization techniques used (e.g. SEM/EDX, Rockwell indenter, and AFM). Chapter 4 described the results of the experiment including discussions of the collected result. Conclusions of the research and recommendations are discussed in the last chapter.